

## SUGAR SUBSTITUTE PREPARED WITH NUTRITIVE AND HIGH-INTENSITY SWEETENERS

### FIELD OF THE INVENTION

The present invention relates generally to high-intensity sweeteners. More particularly, the invention relates to granular sweetener compositions including a mixture of a nutritive sweetener and a high-intensity sweetener, in a form that minimizes separation  
5 of these components during shipping and handling.

### BACKGROUND OF THE INVENTION

Sucralose, 4,1',6'-trichloro-4,1',6'-trideoxygalactosucrose, a sweetener with a sweetness intensity several hundred times that of sucrose, is made from sucrose by replacing the hydroxyl groups in the 4, 1', and 6' positions with chlorine. Synthesis of  
10 sucralose is technically challenging because of the need to selectively replace specific hydroxyl groups with chlorine atoms, while preserving other hydroxyl groups including a highly reactive primary hydroxyl group. Numerous approaches to this synthesis have

been developed. See, for example, U.S. Patents No. 4,362,869; No. 4,826,962; No. 4,980,463; and No. 5,141,860, which are expressly incorporated in this document by reference.

Sucralose may be purified by crystallizing from water to form needle-  
5 shaped crystals, as described for example in U.S. Patents No. 4,343,934; No. 4,950,746, No. 4,977,254; No. 4,980,463; No. 5,136,031; No. 5,498,709; and No. 5,530,106. Many of these crystals typically have a length-to-diameter ("L/D") ratio ranging from about 4:1 to about 10:1, and in some cases even higher. Typically, many such needles are broken, which produces undesirable dust. Nonetheless, at least a significant fraction of the  
10 needles remain that have high L/D values. The high L/D values makes such crystalline sucralose difficult to handle. Among such difficulties is poor flow, which makes the crystalline sucralose difficult to incorporate into formulations with other ingredients. Furthermore, such crystals of sucralose may not be suitable for use in certain consumer products such as sweetener packets for use by a consumer in sweetening food items such  
15 as beverages.

In some such applications, it is desirable for the sweetener to contain some amount of a sugar (typically sucrose, commonly referred to as table sugar) and some amount of a high-intensity sweetener. Sucrose typically has a granular form, however, while sucralose typically has a needle form, as noted above. As a result, mixtures of

these two components may separate to some degree during shipping and handling, resulting in unpredictable levels of sweetening when the consumer dispenses the product.

Thus, in some applications, it is desirable to provide a sweetener that does not separate in this manner, while still maintaining an appearance similar to that of normal table sugar.

5 This result may be of particular importance in formulating certain popular products in which one half teaspoon of a sweetener is made mostly of sucrose with a small amount of a non-nutritive sweetener (such as sucralose), and has the same sweetening power as a full teaspoon of pure sucrose.

There remains a need for granular sweeteners having a combination of  
10 sucrose and sucralose, in which the sucrose and sucralose do not separate during shipping and handling, and resembling common table sugar in appearance and flow characteristics.

### SUMMARY OF THE INVENTION

To meet this and other needs, and in view of its purposes, the present invention provides a sweetener composition, the composition comprising granules  
15 comprising, consisting of, or consisting essentially of (a) a core including a nutritive sweetener, and (b) on the core, a layer including sucralose.

In another aspect, the invention provides a method of making a sweetener composition. The method includes the steps of (a) providing a plurality of granular cores each comprising, consisting of, or consisting essentially of sucrose; and (b) forming on the granular cores a layer comprising, consisting of, or consisting essentially of sucralose.

5 In another aspect, the invention provides a sweetener composition made by the method set forth in the preceding paragraph. It is to be understood that both the foregoing general description and the following detailed description are exemplary, but are not restrictive, of the invention.

#### DETAILED DESCRIPTION OF THE INVENTION

10 For purposes of this application, the term “high intensity sweetener” and “non-nutritive sweetener” are used interchangeably and have the same meaning as those skilled in the art ascribe to such terms, presently or in the future.

The sweetener compositions of the invention include granules, which in turn have a core and a layer deposited on such core. The core includes at least one  
15 nutritive sweetener, and the layer deposited on the core includes sucralose. There may be more than one layer deposited on the core, and any or all of the one or more layers may also include one or more binders and/or other non-nutritive sweeteners.

The nutritive sweetener(s) in the cores of the compositions of this invention may include, as non-limiting examples, sucrose, fructose, invert sugar, dextrose, maltodextrin, sugar alcohols, and combinations of any of these. In one exemplary embodiment, sucrose alone is used, and thus the core consists essentially of sucrose. By “consists essentially of,” it is meant that no other ingredients are purposefully added to the core that change the sweetness profile of the core. Sources of sucrose suitable for use according to the present invention include any commonly available source, such as, for example, beet sugar and cane sugar. They may include white sugar or brown sugar.

No special preparation of the cores need be made, but it may be helpful to use cores of a relatively uniform size in order to provide a more even distribution of sucralose on the cores. A uniform size may be beneficial because the use of cores having a wide distribution of sizes may result in the smaller particles including a higher percentage by weight of sucralose, and thus any settling and separation of small from large particles may result in uneven sweetness within a sample. Commercially available granules from any of a wide variety of sources known in the art may be used, and are typically sieved before use in order to achieve a relatively uniform size distribution. The cores are therefore essentially solid, and are typically of such a size and shape that they resemble common table sugar. A typical (but non-limiting) size distribution for the cores is as follows: no more than 3% retained on a 20-mesh sieve, a minimum of 8%

(cumulative) retained on a 40-mesh sieve, and no more than 10% passing through a 100-mesh sieve.

The layer deposited on the core may contain (other than materials of no essential significance) only sucralose, or it may include sucralose in combination with one or more binders and/or with other non-nutritive sweeteners. Examples of suitable non-nutritive sweeteners that may optionally be added include, but are not limited to, saccharin, acesulfame-K, cyclamate, stevia, neotame, alitame, aspartame, and combinations of such sweeteners.

Binders suitable for use according to the invention include, but are not limited to, nutritive sweeteners, such as those mentioned above, including mixtures of them. Other suitable binders include, as non-limiting examples, maltose, glucose, polyols (e.g., sugar alcohols, such as erythritol and sorbitol), modified food starches, gum, inulin or hydrolyzed inulin, maltodextrin, and combinations of these examples. Typically, the binder will include sucrose, and may contain (other than materials of no essential significance) only sucrose. In one embodiment of the invention, sucralose constitutes between about 30 wt% and about 50 wt% of the layer, with one or more binders making up the balance. Typically, sucralose is present in an amount of between about 0.1 wt% and about 50 wt% of the sweetener composition.

In one embodiment, essentially all of the composition contains (other than materials in trace amounts) only granules having a core with a sucralose-containing layer deposited on the core, with the composition containing between about 0.1 wt% and about 0.50 wt% sucralose, the remainder containing (other than materials of no essential  
5 significance) only sucrose. The resulting sweetener composition has a sweetness equivalence of one-half teaspoon equal to a full teaspoon of sucrose. In another embodiment of the invention, the sweetener composition may contain between about 1.0 wt% and about 50 wt% sucralose, with the rest containing (other than materials of no essential significance) only sucrose. Such a material may be blended with sucrose (in a  
10 ratio depending on the content of sucralose in the material) to provide a product with between about 0.1 wt% and about 0.5 wt% of sucralose overall. Such a blended product is another embodiment of the invention. Other weight percent loadings of sucralose will be apparent to persons skilled in the art, and the invention is not restricted to any particular proportions of sucralose and sucrose on any given granule or fraction of  
15 granules.

The layer including sucralose may be composed of any of a number of formulations. The layer may be provided, for example, by applying a dispersion (which may be a solution) including sucralose in an appropriate volatile carrier, such as water, to the granular cores and evaporating the volatile carrier. Methods of applying such  
20 coatings will be discussed below. In one embodiment, the dispersion that is applied to

the cores contains only sucralose and the carrier, and the layer thus produced contains (other than materials of no essential significance) only sucralose.

In another embodiment of the invention, a binder is included in the sucralose-containing dispersion. There may be other ingredients in the dispersion in  
5 addition to the binder, sucralose, and the carrier, or the dispersion may contain no other added ingredients.

If the layer includes a binder, the sucralose may be dispersed in that binder. For example, the layer may contain (other than materials of no essential significance) only a solid solution of sucralose and binder, by which it is meant that the  
10 sucralose is dissolved in the binder or a component of the binder (if the binder has more than one component), and/or the binder (or a component of the binder) is dissolved in the sucralose. The layer may also, or instead, have particles containing (other than materials of no essential significance) only sucralose dispersed within it. The layer may be essentially continuous, and it may cover essentially the entire surface of a given granule,  
15 or it may include isolated patches of layer composition, or there may be an at least partially interconnected network of regions of layer composition on the cores.

The material in the layer is essentially solid, meaning that there are few or no hollows or voids in the layer, with the obvious exception that the entire layer itself



may be hollow in the sense that it may constitute a continuous shell around the core.

Sweetener compositions according to the invention, as a result of having a structure as described above, typically have a bulk density approximately equal to that of common

table sugar. Thus, in at least some embodiments, especially those based on sucrose as the  
5 nutritive sweetener core material, the bulk density may be in the range of about 0.7 to 0.9, typically between 0.8 and 0.86.

As noted above, sweetener compositions according to the invention include granules with a core and a layer. The composition may contain (other than

materials of no essential significance) only such granules, or it may include the granules

10 mixed with other materials. Such other materials may include non-sweet diluents and/or

nutritive sweeteners. In one embodiment of the invention, the granules are mixed with a

nutritive sweetener including, or containing (other than materials of no essential

significance) only sucrose. In one exemplary embodiment of the invention, the sweetener

composition includes about 10 wt% of a granular material including a core and a layer on

15 the core, with the rest of the composition containing (other than materials of no essential

significance) only granules of uncoated sucrose. In this embodiment, the sucralose in the

granules having a core and a layer constitutes between about 1 wt% and 5 wt% of those granules.

In one embodiment of the invention, the layer contains sucralose and sucrose, which acts as a binder, and the core is sucrose as well. Such a composition may have storage stability, as measured by a 14-day accelerated aging test, greater than that of compositions in which the sucralose is not provided in a layer on a sucrose core. The  
5 term "14-day accelerated aging test," as used for the purposes of this invention, is described in detail in the Examples section, and measures the color of the material according to the Hunter Lab<sup>®</sup> method, which is well known in the art. Sweetener compositions according to the invention have good stability as indicated by this test, and may for example have a Hunter "a" value no greater than 0.2 units, more typically no  
10 greater than 0.1 units, higher than the Hunter "a" value of the composition before aging. They may have a Hunter "b" value that is no greater than 1.0 units higher than the "b" value of the composition prior to aging.

Without wishing to be bound by any particular theory or explanation, it is believed that the enhanced color stability of sweetener compositions according to the  
15 invention may result from the dispersion of the sucralose into small domains in the layer residing on the cores. These small domains may be small particles containing (other than materials of no essential significance) only sucralose, or the domains may be of a molecular size, in other words, a solid solution of sucralose in a binder component, or vice versa. It is believed that, for either or both of these reasons, dispersion or dilution of  
20 the sucralose results in increased stability of the sweetener composition.

The layer including sucralose and (optionally) a binder may be formed on the cores by any of a number of methods known in the art. Such methods include, as non-limiting examples, spray-drying and fluidized bed drying. One particularly suitable method of forming the layer is by pan-coating, which may be used in either batch mode or continuous mode. Equipment suitable for pan-coating of granules such as those of the present invention is well-known in the pharmaceutical compounding art, and is available from a variety of manufacturers. A typical machine has a rotating pan in which the granular cores tumble. A spray nozzle is typically placed through an opening in the drum, and a mixture of the sucralose-containing layer composition in a volatile carrier is sprayed onto the granular cores. A wave is created as the pan turns, and the granules tumble in a wave form. The coating material slowly builds up on the granules, which are dried to form the layer.

The coating operation may be performed in a single step, with drying being performed either concurrently or subsequently to the spraying on of the aqueous mixture. Typically, a series of coating/drying cycles is performed, until the desired layer weight is produced. Optionally, particles including sucralose may be introduced after spraying but before drying is complete, so that the particles adhere to the granules. This may reduce the time required to provide the desired amount of sucralose, by reducing the drying time per unit of sucralose added. Drying may be performed by any mechanism known in the art, including for example the application of a stream of air or other gas,

such as nitrogen, and/or warming the contents of the rotating pan. For example, it has been found suitable to warm the contents of the rotating pan to between about 38°C and 93°C, more typically between 52°C and 66°C, because drying within this temperature range has been found to be helpful in providing a product with good accelerated aging  
5 stability.

In one embodiment of the invention, the sucralose to be incorporated in the layer is added in the form of an aqueous mixture, optionally containing other volatile carriers. Such carriers may be one or more of any of a number of suitable volatile materials, with non-limiting examples being methanol, ethanol, isopropyl alcohol, and  
10 ethyl acetate. Typically, water is the only carrier and the aqueous mixture also contains sucrose as a binder. In some embodiments, the binder contains (other than materials of no essential significance) only sucrose.

In one embodiment of the invention, substantially all of the sucralose to be used in forming the layer is dissolved in the aqueous mixture. Such a mixture may for  
15 example include between 20 wt% and 60 wt% of water, between 15 wt% and 55 wt% of sucrose, and between 15 wt% and 35 wt% of sucralose. Typically, the mixture includes between 35 wt% and 45 wt% of water, between 31 wt% and 41 wt% of sucrose, and between 18 wt% and 28 wt% of sucralose.

### Examples

The following examples are included to more clearly demonstrate the overall nature of the invention. These examples are exemplary, not restrictive, of the invention.

5     *Example 1*

A sweetener composition according to the invention was prepared as follows. A mixture of 23.2 parts by weight of sucralose, 54.2 parts by weight of a 67.7 wt% aqueous sucrose solution, and 22.6 parts by weight of water was mixed at 70°C until all components were dissolved. The resulting coating composition contained 23.2 wt% of  
10     sucralose, 36.8 wt% of sucrose, and 40.0 wt% of water.

Extra fine sugar (sucrose) was sieved through a 52T screen and charged to a 48-inch Stokes coating pan. The contents of the coating pan were heated to approximately 54°C, and several applications of coating composition were applied by a sprayer operated at an average spray pressure of 84 psi, using a No. 30 spray tip equipped  
15     with an inline filter. After each application, the product was heated until dry (which was indicated by an operator squeezing the product in a fist, to make sure it did not clump) before the next coating was applied. During the coating and drying operations, a stainless steel hoop was used to agitate the bed of granular material. During the coating process,

the average bed temperature was approximately 57°C. The resulting product was sieved through an 18T sieve and had a sucralose content of 2.2 wt%. This concentrated product was dry-blended in a weight ratio of 1 part out of 10 with extra fine sugar (sieved as described above) using a rotary tote blender to provide a product having a sucralose  
5 content of 0.22 wt%. This material had a composition such that one half teaspoon had the sweetness equivalence of a full teaspoon of sucrose.

*Example 2 (Comparative)*

A sweetener composition including a simple mechanical mixture of sucrose and sucralose was prepared as follows. A composition containing 0.22%  
10 micronized sucralose and 99.78% extra fine sugar was blended for 5 minutes using a TURBULA<sup>®</sup> Shaker-Mixer – Model T2F, available from Glen Mills Inc. of Clifton, NJ. Accelerated aging of the samples prepared in Examples 1 and 2 was performed as follows. The sweetener composition was maintained at 50°C and sampled after a given number of days. The color of a dry sample of the composition was evaluated in a 10-mm  
15 cuvette by the Hunter Lab<sup>®</sup> method. According to this method, samples having an “a” value greater than zero have a net red color component; those samples with a negative “a” value have a net green color component. Similarly, samples having a Hunter “b” value greater than zero have a net yellow color component; those with a negative “b” value have a net blue color component. Samples having both “a” and “b” values equal to zero

are colorless. Samples having high “L” values are brighter than those with low values.

The results of color testing of the sweeteners prepared in Examples 1 and 2 after accelerated aging for the number of days indicated are shown in the tables below, in which each data point represents an average of nine color determinations.

#### Example 1

Measurement	Day 0	Day 3	Day 4	Day 5	Day 6	Day 7	Day 14
L (Avg.)	90.77	91.00	91.48	90.64	90.31	89.66	91.54
a (Avg.)	-0.36	-0.33	-0.35	-0.34	-0.38	-0.34	-0.33
b (Avg.)	3.85	3.93	3.82	4.18	4.01	3.91	4.46

#### Example 2

Measurement	Day 0	Day 3	Day 4	Day 5	Day 6	Day 7	Day 14
L (Avg.)	90.17	89.91	90.59	89.48	89.48	89.42	89.57
a (Avg.)	-0.52	-0.51	-0.43	-0.40	-0.40	-0.33	0.26
b (Avg.)	5.84	6.17	6.36	6.26	6.08	6.16	7.35

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As can be seen by comparing the accelerated aging results obtained with Examples 1 and 2, increases in “a” and “b” values as a result of 14 days of accelerated aging were noticeably less for the layer-and-core sweetener composition according to the invention (Example 1) than for the simple mechanical mixture of sucrose and sucralose (Example 2).

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Although the invention is illustrated and described above with reference to specific embodiments, the invention is not intended to be limited to the details shown.

Rather, various modifications may be made in the details within the scope and range of equivalents of the claims and without departing from the invention. It is expressly intended, for example, that all ranges broadly recited in this document include within their scope all narrower ranges which fall within the broader ranges.